AMENDMENT NO. 2 NOVEMBER 2022

TO

IS 3470 : 2017 HEXANE, FOOD GRADE — SPECIFICATION

(Second Revision)

(*Page* 11, *Annex* F) — Substitute existing Annex with the following:

ANNEX F

[*Table* 1, *Sl No.* (x)]

DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS

F-1 SCOPE

This method prescribes the test for determining the polycyclic aromatic hydrocarbons in food grade hexane sample.

F-2 GENERAL INSTRUCTIONS

All glassware should be scrupulously cleaned to remove all organic matter such as oils, grease, detergent residue, etc. Examine all glassware, including stopper and stopcocks, under ultraviolet light to detect any residual fluorescent contamination. As a precautionary measure, all glassware may be rinse with purified iso-octane immediately before use. No grease is to be used on stopcocks or joints. Because some of the polynuclear hydrocarbons sough in this test are very susceptible to photo-oxidation, the entire procedure is to be carried out under subdued light.

F-3 APPARATUS

F-3.1 Separatory Funnel

250 ml capacity equipped with polytetrafluroethylene (PTFE) polymer stopcocks.

F-3.2 Spectrophotometric cells

Fused quartz cells, optical path length in the range of 5.0 ± 0.005 cm; also for checking spectrophotometer performance only, optical path length in the range 1.0 ± 0.005 cm. With distilled water in the cells, determine any absorbance difference.

F-3.3 Spectrophotometer

Spectral range 200-450 nm with spectral slit width of 2 nm or less; under instrument operating conditions for these absorbance measurements, the spectrometer shall also meet the following performance requirements:

Absorbance repeatability: ± 0.01 at 0.4 absorbance
Absorbance accuracy: ± 0.05 at 0.4 absorbance
Wavelength repeatability: ± 0.2 nm
Wavelength accuracy: ± 1.0 nm

F-4 REAGENTS

F-4.1 *iso*-Octane (2,2,4-Trimethyl pentane)

UV spectroscopic grade. If necessary, iso-octane may be purified by passing through a column of activated silica gel (Grade 1) or by distillation.

F-4.2 Dimethyl Sulphoxide (DMSO) — UV Spectroscopic Grade

F-4.3 *n*-Hexane — UV Spectroscopic Grade

F-4.4 Naphthalene — Analytical Reagent Grade

F-5 PROCEDURE

F-5.1 Preparation of Standard (Reference) Solution of Naphthalene and Measurement of Absorbance

Prepare a standard (reference solution) of naphthalene

Price Group 1

in iso-octane containing 7.0 mg of naphthalene per litre. Measure the absorbance of the standard (reference solution) at 275 nm using iso-octane as blank in 5.0 cm quartz cell.

F-5.2 Determination of UV Absorbance of the Test Solution

F-5.2.1 Add 75 ml of n-hexane in a 250 ml separating funnel. Add 5 ml of dimethyl sulphoxide (DMSO). Shake vigorously for 1 min and allow to stand until two clear layers are formed. Collect the clear lower layer and use as blank to measure the absorbance of the test solution in 5.0 cm quartz cell.

F-5.2.2 Take two 250 ml separating funnels. Transfer 75 ml of sample to the first 250 ml separating funnel and add 75 ml of n-hexane. Mix well and add 15 ml of dimethyl sulphoxide (DMSO). Shake vigorously for 1 min and allow to stand for minimum 60 min or until two clear layers are formed at approximately 25 ± 2 ºC.

F-5.2.3 Transfer the separated clear lower layer to a second separating funnel, add 6 ml of n-hexane using a pipette and shake the mixture vigorously. Allow to stand for minimum 30 min or until two clear layers are formed. Separate the lower layer and measure the absorbance over the range of 260 nm to 420 nm using iso-octane as blank.

F-5.3 At any wavelength in the range 260 nm to 420 nm the absorbance of the test solution should not exceed one-third of the absorbance of the standard (reference solution) at 275 nm.

(PCD 03)

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